THE MODELLING OF PROCESSING CONDITIONS FOR POLYPROPYLENE-NANOCLAY INTEGRAL HINGES AT HIGH HEAT EXPOSURE

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ABSTRACT

This research is about generating models of injection moulding processing conditions, towards quality performance of polypropylene-nanoclay integral hinges, exposed to high heat temperature. The assessment of hinges’ quality performance analyses was translated as the signal to noise ratio values for ultimate tensile strength, shrinkage and warpage. This research had adopted Taguchi Optimisation Method, to optimise the processing conditions, to generate the regression models and to construct master curves for quality performance prediction based on nanoclay content. According to the results, 18 regression models have been successfully generated. 3 types of master curves have been constructed based on the produced models with the specific nanoclay content. Additionally, the quality performance of the integral hinges was extended to high heat exposures, and the additional of nanoclay had produced a significant advancement in the injected mould samples. Validation test has been carried out towards the regression model with most of the models have produced good predictions of quality performances. The novelty of this research is the correlations between the optimum injection moulding processing conditions with the precise range of shrinkage, warpage and ultimate tensile strength. The correlations were simplified in the form of regression models and master curves. These models and master curves were recommended as references and a prediction method, specifically for polypropylene-nanoclay integral hinges manufacturing and design process. These findings will lead to wider and optimum applications of thin layer parts and components such as packaging products; as well as other manufacturing field such as artificial human parts development and building appliances.
ABSTRAK

Kajian ini adalah mengenai penjanaan model keadaan pemprosesan bagi proses acuan suntikan; terhadap prestasi kualiti engsel bersepadu polipropelina-tanah liat nano yang terdedah pada suhu tinggi. Penilaian analisis prestasi kualiti ini diterjemahkan melalui nilai-nilai nisbah isyarat terhadap hingar bagi kekuatan tegangan muktamad, pengecutan dan perlengkungan. Kajian ini telah menggunakan pakai Kaedah Pengoptimuman Taguchi, untuk mengoptimumkan keadaan pemprosesan, untuk menjana model regresi dan untuk membangunkkan keluk induk bagi prestasi kualiti berdasarkan kandungan tanah liat nano. Berdasarkan kepada keputusan yang diperolehi, 18 model regresi telah dijana. 3 jenis keluk induk telah dibangunkan berdasarkan model yang dihasilkan, mengikut kandungan tanah liat nano yang khusus. Sebagai tambahan, prestasi kualiti engsel bersepadu ini diperluaskan kepada pendedahan terhadap suhu yang tinggi, dengan penambahan tanah liat nano yang mampu menghasilkan kemajuan nilai terhadap sampel acuan suntikan. Ujian pengesahan telah dijalankan ke atas model regresi dan kebanyakan model menghasilkan ramalan prestasi kualiti yang baik. Keunikan penyelidikan ini adalah korelasi antara keadaan pemprosesan acuan suntikan yang optimum dengan julat nilai yang jitu bagi pengecutan, perlengkungan dan kekuatan tegangan muktamad. Kolerasi ini telah diterjemahkan dalam bentuk model regresi dan keluk induk, yang mana hasil dapan ini sangat dicadangkan sebagai rujukan dan kaedah ramalan, khusus untuk proses pembuatan dan rekabentuk engsel bersepadu dari polipropelina-tanah liat nano. Penemuan ini akan membawa kepada aplikasi yang lebih luas dan optimum bagi pembuatan bahagian-bahagian dan komponen berlapisan nipsis, seperti produk pembungkusan dan juga bidang pembuatan lain seperti pembangunan anggota tiruan manusia dan peralatan pembinaan.
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LIST OF SYMBOLS AND ABBREVIATIONS

$^0C$ - Degree Celsius
2D WAXS - Two Dimension Wide Angle X-ray Scattering
ABS - Acrylonitrile-Butadiene-Styrene
AFM - Atomic Force Microscopy
ANN - Artificial Neural Network
ANOVA - Analysis of Variance
BP - Back Propagation
CAD - Computer Aided Design
CAE - Computer Aided Engineering
CBR - Case Based Reasoning
CEC - Cation Exchange Capacity
CNC - Computer Numerical Control
DMA - Dynamic Mechanical Analysis
DSC - Differential Scanning Calorimetry
EVA - Ethylene Vinyl Acetate
FEM - Finite Element Method
FKMP - Faculty of Mechanical and Manufacturing Engineering
FT - Filling Time
FTIR - Fourier Transfer Infra Red
G - Grams
G' - Storage Modulus
GA - Genetic Algorithm
H - Height
HDPE - High Density Polyethylene
HRS - Hot Runner System
HSC - High Strength Concrete
iPP - Isotactic Polypropylene
\[ L \] - Length
\[ \bar{L} \] - Average Length
\[ L_c \] - Length of Actual Mould Cavity
\[ LRM \] - Linear Regression Method.
\[ MD \] - Machine Direction
\[ \text{min} \] - Minute
\[ mm \] - Millimetre
\[ MPa \] - Mega Pascal
\[ MSD \] - Mean Squared Deviation
\[ MT \] - Melt Temperature
\[ MWNT \] - Multi Walled Nano Tubes
\[ ND \] - Normal Direction
\[ \text{o-MMT} \] - Organo-Montmorillonite
\[ OLS \] - Ordinary Least Squares
\[ P-V-T \] - Pressure-Volume-Temperature
\[ PC/ABS \] - Polycarbonate / Acrylonitrile–Butadiene-Styrene
\[ PCA \] - Principal Component Analysis
\[ PP \] - Packing Pressure
\[ PP-EP \] - Polypropylene-Ethylene Propylene
\[ PP-g-AA \] - Polypropylene-grafted-Acrylic Acid
\[ PP-g-MA \] - Polypropylene–grafted- Maleic Anhydride
\[ Q1@60^\circ C \] - Quality Performance for the first formulation for the exposure condition at 60\(^\circ\)C
\[ Q1@70^\circ C \] - Quality Performance for the first formulation for the exposure condition at 70\(^\circ\)C
\[ Q1@RT \] - Quality Performance for the first formulation for the exposure condition at Room Temperature
\[ QP \] - Quality Performance
\[ R-Sq \] - Correlation Coefficient
\[ R-Sq (adj) \] - Correlation Coefficient (Adjusted)
\[ S \] - Second
\[ S \] - Shrinkage
\[ S/N \] - Signal to Noise
$S/N_{QP}$ - Signal to Noise for Quality Performance
$S/N_{S}$ - Signal to Noise for Shrinkage
$S/N_{UTS}$ - Signal to Noise for Ultimate Tensile Strength
$S/N_{Z}$ - Signal to Noise for Warpage
$SA$ - Simulated Annealing
$SEM$ - Scanning Electron Microscopy
$SIM$ - Sequential Injection Moulding
$SIRIM$ - Standards and Industrial Research Institute of Malaysia
$SS$ - Screw Speed
$T_{ambient}$ - Ambient Temperature
$T_{mould}$ - Mould Temperature
$t_a$ - Average Thickness
$TD$ - Transverse Direction
$TEM$ - Transmission Electron Microscopy
$TGA$ - Thermal Gravimetric Analysis
$TS$ - Transmission Spectroscopy
$UTM$ - Universal Testing Machine
$WAXS$ - Wide Angle X-ray Scattering
$wt.\%$ - Weight Percentage
$XRD$ - X-Ray Diffraction
$Z$ - Warpage
$\alpha$ - Coefficient of Thermal Expansion
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CHAPTER 1

INTRODUCTION

In this chapter, discussion about research background, problem statements, objectives and scope of study in this research have been presented. The expected result, which is the novelty of this research also, was stated at the end of this chapter.

1.1 Background study

This research is about modelling of injection moulding processing conditions toward the performance of functional polypropylene integral hinges samples with the additional of nanoclay Cloisite-20A, exposed to high heat temperature. The research started by performing injection moulding simulation and practical work; in order to verify and validate the developed mould for test the samples. The mould test sample was used to optimise the processing conditions towards shrinkage, warpage and ultimate tensile strength, with exposure to high heat environment. These properties analysis are very important in the manufacturing industry, because the characterisation of functional integral hinges was very useful in various thermoplastic component designs and manufacturing processes. The findings of this research, in the form of the regression model and developed the master curves, can lead to production of better quality, with longer life span of functional integral hinges. The model and curves were very useful as references, for further applications and improvement with wide potential in the manufacturing of plastic parts and components. Moreover, the additional value of this research was the usage of nanoclay Cloisite-20A as the nano size filler in the
polymer nanocomposites system was rectified to withstand exposure to the high heat environment.

1.2 Problem statement

The manufacturing of thin wall components with integral hinges is crucial for the several plastic industries due to the thinner components permit considerable improvement of environmental impact, beneficial effects on the reduction of fuel consumption and overall weight savings. Additionally, the decrease in thickness allows significant reduction in production costs because shorter cycle times and less material consumption. Particularly, thin wall plastic products relate to smaller and lighter parts; should be able to withstand the daily usage, and at the same time maintaining their aesthetic appearance. These features are critical in the realization of automotive interior components that must maintain high quality look and feel throughout the life cycle of the vehicle (Spina, 2004).

These intricate parts in the plastic industry, comes with toughest quality requirements, could be achieved through injection moulding technology (Nardin et al., 2002). Due to its ability to produce multifaceted shape plastic parts with good dimensional accuracy and very short cycle times, injection moulding has become one of the processes that is greatly preferred in manufacturing industry (Bozdana & Eyercioglu, 2002). These injected moulded parts, however, are very prone to defects. To avoid such quality control problems, it is desirable to successfully predict the optimum processing conditions, such as pressures, temperatures, and times (Hill, 1996). Any change in these variables can affect the process stability and the quality of the manufactured parts. Unfortunately, it has been found difficult to control and adjust simultaneously between the processing conditions and the properties of the product; and there is no single set of rules to designate which parameters to use in order to manufacture consistently a part with no defects (Dumitrescu et al., 2005). Furthermore, failures or damages of these parts become more frequent when it exposed to high temperature environment.

In order to solve the problems, optimising the processing conditions through several techniques becomes one of the promising solutions. The optimisation methods, such as Principal Component Analysis (Fung & Kang, 2005) ; Neuro Fuzzy Model (Antony & Anand, 2006) ; Gaussian Process Approach with Genetic Algorithm (Zhou
& Turng, 2007), Grey Relational Analysis (Khan et al., 2010) and Taguchi (Mehat & Kamaruddin, 2011) have been proven as a good solution in finding the suitable injection moulding processing condition.

Additionally, the utilisation of fillers in polymer composites also a possible solution, whereby the fillers such as nanoclay could improve the mechanical properties as well as the fire retardant behaviour. However, too many fillers with wrong processing condition might lead back to poor quality and component performance (Wang, 2012; Yuan et al., 2008).

Therefore, this research proposes to solve the problem of thermoplastic integral hinges parts design and processing conditions, by introducing a suitable model and master curves as the reference to predict the properties of polypropylene integral hinges with the additional of nanoclay Cloisite-20A. By transforming the pristine polypropylene into polymer nanocomposites, with the right clay content and processing conditions, the integral hinges were expected to sustain its function; even when it was exposed to the high heat environment.

1.3 Objectives

The objectives of this research are:

a) To develop mould for producing the integral hinges test’s sample.

b) To optimise the processing conditions by adopting the Taguchi Optimisation Method.

c) To generate regression model base on the optimised processing condition of polypropylene-nanoclay integral hinges with the responses of quality performance, exposed to 60°C to 70°C.

 d) To construct master curves based on the regression model for quality performance versus clay content for integral hinges.

1.4 Scope of study

The scopes of this study are as stated:

a) The raw material selected is polypropylene, a type of homopolymer, Titan Pro 6331, from Titan Petchem (M) Sdn. Bhd. The compatibilizer is a functionalized
polypropylene-grafted-maleic anhydride (PP-g-MA), containing 1 wt. % of maleic anhydride (OREVAC C100).

b) The additional material to develop these polymer nanocomposites is nanoclay Cloisite-20A, varies from 0 wt. %, 1 wt. %, 2 wt. %, 3 wt. %, 4 wt. % and 5 wt. %, obtained from Southern Clay, Inc. US, courtesy from Wilbur-Ellis Company, Connell Bros. Company (Malaysia) Sdn. Bhd.

c) The performance of polypropylene integral hinges shall be translated through shrinkage, warpage and ultimate tensile strength. The test conducted for the manufactured samples shall adopt ISO standards.

d) The mould was made from AISI D2 cold work tool steel. Evaluation shall be made to choose the best mould design to define runner size and gate location.

e) The expected result shall focus more on generating the regression model that shall be fitted in the master curves, after getting the optimised processing condition by using the Taguchi Optimisation Method.

f) The chosen processing condition were screw speed, melt temperature, injection pressure and filling time.

1.5 Expected result

In this research, a mould for preparing test samples specifically representing an integral hinge component was produced. These samples were made by using the mould that had been developed using simulation. The actual mould was fabricated and actual injection moulding was carried out to validate the simulation findings. The novelty of this research is the correlations between optimum injection moulding processing conditions and the quality performances of the products, exposed to the high heat environment, which were translated in the form of regression models. Master curves have been constructed for quality performance versus the nanoclay content, based on the regression models. The model and master curves can be used as references specifically for polypropylene-nanoclay integral hinges manufacturing and design process, with the extend condition in high heat environments. The additional of nanoclay shall be the additional advantage, whereby with the right nanoclay content, the quality performance of integral hinges was improved.
CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter is about the past works related to this project. It summarizes several previous researches which were related to this project. Previous research findings about the optimisation of injection moulding processing condition and research about polypropylene-nanoclay were included in this chapter. Research on the application of polypropylene, especially at high heat exposure was also been stated at the end of this chapter.

2.1.1 Injection moulding processing conditions

In general, injection moulding is a process that involves hot, injection moulded molten polymer which is heated with a highly plastic state; and then injected automatically by a screw with the support of hydraulics actuator, under high pressure into a mould cavity where it solidifies into cooled mould where the molten polymer will follow the final shape of the mould. The moulded part is then removed from the cavity. The advantage of this process is, that it may produce discrete components that are almost net shape and significant economies of mass production. The production cycle time is typically in the range of 10-30 seconds, for small components and longer time is not uncommon for large parts. Moreover, the mould may contain more than one cavity, therefore multiple mouldings are produced for each cycle and it also increases the cycle time as well. An injection moulding machine usually consists of an injection unit
Intricate and complex designs/dimensions are possible with injection moulding. In these cases, the challenge is to design and fabricate a mould that has the same geometry as the original component and which also easy for part removal. Part sizes can be in the range from about 50 grams up to about 25 kilograms, whereby the bigger parts represented by components such as refrigerator doors and automobile bumpers. The mould is the special tooling in injection moulding, because not only it determines the part shape and size, it also contributes a lot towards parts quality. For large, complex parts, the mould can be expensive, depending on the type of material and the machining time. For small parts, the mould can be built to contain multiple cavities, which is also making the mould expensive. Thus, injection moulding is economical and provide a good return of investment only for mass production (Groover, 2007).

### 2.1.2 Mould for injection moulding

Mould in injection moulding usually is in the form of cavity whereby the molten polymer shall be injected and solidified in this particular part. The surface of this tool will act as heat exchanger when the injected material solidifies with contact or for cooling the moulded component. The most common cold runner in plastic injection moulding tooling is a two-plate, cold runner mould at horizontal injection moulding. With thermoplastic materials, a cold runner mould refers to a mould in which the
runner is cooled, solidified, and ejected with the moulded part during each moulding cycle. This tooling becomes common due to its simplicity, least expensive, easy to construct and the easiest to operate with less maintenance as compared with the hot runner mould. This type of mould consists of two halves; which is stationary and movable half; fastened to the two platens of the moulding machine’s clamping unit. When the clamping unit opened, the two mould halves open. The function of movable half is to eject finish or desired part (Osswald et al., 2008).

Most of the mould consists of the cavity, sprue, runner, gates, injection system and cooling system. These elements are very important in ensuring the melt polymer distributed uniformly. The details about these elements are stated as below (Osswald et al., 2008).

a) Cavity - the feature which usually formed by removing metal from the mating surfaces of the two halves. Moulds can contain a single cavity or multiple cavities to produce more than one part in a single shot.

b) Sprue /distribution channels – This element leads the melt polymer from the nozzle into the mould. The sprue is in the form of carrot and act as an inlet channel to transfer molten material from the heating chamber into the runner system.

c) Runner - the function is to lead the melt polymer from the sprue to the multiple cavity moulds. Runner also acts as channels to connect the sprue bush to the cavity gates. There are two types of runner system-cold and hot. Cold runners are ejected with the part are trimmed after mould removal. The advantage of cold runner is lower mould cost. The hot runner keeps the polymer at or above its melt temperature. The material stays in the runner system after injection for the next injection.

d) Gates- Is a part that constricts the flow of plastic into the cavity. It prevents material from flowing out of the cavity when the injection pressure removed. There are one or more gates for each cavity in the mould.

e) Ejection system- A system required to eject the moulded part from the cavity at the end of the moulding cycle. Ejector pins built into the moving half of the mould usually accomplish this function. The cavity is divided between the two mould halves in such a way that the natural shrinkage of the moulding causes the part to stick to the moving halt. When the mould opens, the ejector pins push the part out of the mould cavity.
f) Cooling system- This system consists of an external pump connected to passageways in the mould, through which water is circulated to remove heat from the hot plastic. Air must be evacuated from the mould cavity as the polymer rushes in. Much of the air passes through the small ejector pin clearances in the mould. In addition, narrow air vents are often machined into the parting surface; only about 0.03 mm deep and 12 to 25 mm wide, these channels permit air to escape to the outside but are too small for the viscous polymer melt to flow through.

Besides of these elements, there are other crucial factors that need to be considered specifically for thin-layered product. The factors are cycle time, gate locations and cavity condition. Cycle time is largely dependent on section thickness, machine conditions, heating capacity and injection capacity. The overall cycle time can vary from apply five seconds for thin articles to 60 seconds or more for thick articles. As for injection moulding hinges, it is important that the flow front cross the thin section at one instant. Gate location must provide balanced fill. The substantial pressure drop occurs when the flow front crosses the hinge, resulting in an increase of shrinkage rate. This may require adjustment of cavity dimensions to ensure proper fit of mating halves. The flow through the hinge will generate additional shear heating requiring additional local cooling. If the mating halves require two gates for filling and packing, they must be designed and developed to locate the weld line away from the hinges (Bauccio et al., 1994).

2.1.3 Simulation in injection moulding

With plastics gaining more and more ground in engineering applications, there was a critical demand on the quality of injected moulded parts. To satisfy these demands, software was manipulated to achieve the outstanding level of part design, mould design, machining of cavities, and part mouldings (Beaumont et al., 2002). Correct control of the processing condition usually plays major roles in achieving good quality, whereby this parameter settings are usually quantified either based on statistical experimental methods, computer aided simulations, or through operators’ experiences. Table 2.1 shows a review of several studies which have manipulated the effectiveness of numerical simulation.
Many studies have validated the effectiveness of using numerical simulations as the input for producing good quality products through the actual injection moulding process (Beaumont et al., 2002; Othman et al., 2012). To quote a few examples, several researchers managed to discover several great issues through injection moulding simulation, such as the mould ability index (Kumar et al., 2002), effects of flow rate and temperature (Pantani et al., 2005), pressure (Pantani et al., 2007), determination of part thickness and filling ratio (Song et al., 2007), gate design (Shen et al., 2008) and cooling system (Hassan et al., 2010).

Table 2.1: A review of simulation in injection moulding processes

<table>
<thead>
<tr>
<th>Author (Year)</th>
<th>Inputs</th>
<th>Output</th>
<th>Findings</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kumar et al., (2002)</td>
<td>Flow rate, melt-mould thermal contact resistance coefficient</td>
<td>Injection mouldability index (IMI)</td>
<td>The simulation results for the non-isothermal filling indicate physically realistic trends and lend insight into various important aspects of mould-filling including frozen skin layer.</td>
</tr>
<tr>
<td>Pantani et al., (2005)</td>
<td>Distribution of crystallinity degree, molecular orientation and crystals structure and dimensions.</td>
<td>The effects of both injection flow rate and mould temperature are analysed.</td>
<td>Final morphological characteristics of the samples Comparison with the predictions of a simulation code developed at the University of Salerno for the simulation of the injection moulding process</td>
</tr>
<tr>
<td>Pantani et al., (2007)</td>
<td>Packing pressure on morphology, distribution</td>
<td>Microstructure formation. Effects of pressure on crystallization kinetics</td>
<td>Simulation of moulding tests by means of a code developed at the University of Salerno</td>
</tr>
<tr>
<td>Song et al., (2007)</td>
<td>Injection rate, pressure melts temperature &amp; metering size</td>
<td>Part thickness and filling ratio</td>
<td>Part thickness is the decisive parameter to the moulding, metering size and injection rate are the principal factors in the moulding process, and accelerating injection rate can bring a great increase in the filling ratio.</td>
</tr>
<tr>
<td>Shen et al., (2008)</td>
<td>Processing phenomenon of electronic dictionary battery covers in thin-walled injection moulding</td>
<td>The gate design of thin-walled injection moulding.</td>
<td>Numerical simulations of three-dimensional injection moulding for thin-walled product. Gate for single point of two sides is suitable for thin-walled injection moulding by numerical simulation.</td>
</tr>
<tr>
<td>Hassan et al., (2010)</td>
<td>A Cross type rheological model depending on the temperature and pressure is assumed for the polymer material.</td>
<td>Effect of the cooling system on the shrinkage rate The compressibility behaviour of polymer material</td>
<td>Good agreement between the numerical solution and those of the literatures. The position of the cooling channels has a great effect on final product temperature and the shrinkage rate distribution</td>
</tr>
</tbody>
</table>
In terms of software utilisation, the types of software which have been used as a tool in injection moulding research, such as MoldFlow (Cheng et al., 2009; Harmia & Friedrich, 1997; Koszkul & Nabialek, 2004; Tatara et al., 2006), CMOLD (Sridhar & Narh, 2000; Yeung & Lau, 1997) and CadMould (Othman et al., 2012). This software has been produced good findings for future exploration, whereby by manipulating the advanced Computer Aided Engineering (CAE), this tool was programmed to predict the desired responses of injected moulding product. A summary of injection moulding simulation, consisting of the area of studies and the types of software used in research has been compressed in Table 2.2

Table 2.2: Summary of simulation area of studies and software

<table>
<thead>
<tr>
<th>Author (Year)</th>
<th>Area of study in injection moulding</th>
<th>Software</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shen et al., (2001)</td>
<td>Length to thickness ratio of injection moulding products</td>
<td>MoldFlow</td>
</tr>
<tr>
<td>Nardin et al., (2002)</td>
<td>Optimisation of part-mould technology system</td>
<td>DIPSIDOS</td>
</tr>
<tr>
<td>Shamsudin et al., (2012)</td>
<td>Processing condition, weld lines</td>
<td>CadMould 3D-F</td>
</tr>
</tbody>
</table>

Based on these findings, it is proven that by using CAE tools such as injection moulding simulation, good results can be obtained with minimal cost and time consumption. It has been proven that CAE has been successfully used in the simulation of the injection moulding process, since it provides designers/engineers with visual and numerical feedback of the polymer behaviour and eliminates the traditional trial and error approach for optimising. Proper interpretation of the results from simulation can help in selecting a suitable material; reduce cycle time and costs on mould alteration (Chen, et al., 2009). Good results from simulation also can be used as reference in designing the real mould and predicting the output based on the selected design.
2.1.4 The effect of processing conditions

The injection moulding process is a cyclic process. This process consists of several activities such as plastication; injection; packing; cooling, ejection and quality control appliances. Having preliminary knowledge about these activities is vital hence the present day competitive conditions that forced the manufacturing industries to produce faster and cheaper product with a higher quality, such as minimum warpage, sink marks, etc. Therefore, several important processing conditions for each stage of the process may influence the quality of injection moulded plastic products. The processing conditions such as melt temperature, mould temperature, injection pressure, injection velocity, packing pressure and cooling time, need to be optimised in order to achieve good results in injection moulding processes (Ozelik & Erzurumlu, 2006).

To describe the influence of processing conditions towards injection moulding products, for instance, based on Table 2.3 by increasing the injection pressure or increasing mould temperature can produce shrinkage (Bryce, 1996). Meanwhile, lowering back pressure, as well as lowering melt temperature can achieve less degradation. These examples demonstrate that the basic moulding parameters do work closely together and that changing a processing condition in one area may affect a value of any property in another area. By understanding this relationship, it is possible to minimize the number of adjustments required when it is necessary, making a correction due to an unexpected change in some variable of the process (Bryce, 1996).

Table 2.3: Parameter change versus property effect (Bryce, 1996)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Properties Effect</th>
</tr>
</thead>
<tbody>
<tr>
<td>Injection pressure</td>
<td>- increased: Less shrinkage, higher gloss, less wrap, harder to eject</td>
</tr>
<tr>
<td></td>
<td>- decreased: More shrinkage, less gloss, more wrap, easier to eject</td>
</tr>
<tr>
<td>Back pressure</td>
<td>- increased: Higher density, more degradation, fewer voids</td>
</tr>
<tr>
<td></td>
<td>- decreased: Lower density, less degradation, more voids</td>
</tr>
<tr>
<td>Melt temperature</td>
<td>- increased: Faster flow, more degradation, more brittle, flashing</td>
</tr>
<tr>
<td></td>
<td>- decreased: Slower flow, less degradation, less brittle, less flashing</td>
</tr>
<tr>
<td>Mould temperature</td>
<td>- increased: Longer cycle, higher gloss, less wrap, less shrinkage</td>
</tr>
<tr>
<td></td>
<td>- decreased: Faster cycle, lower gloss, greater warp, higher</td>
</tr>
</tbody>
</table>
More often than not, depending on the cavities that being filled, high injection speed is used for this material because fast filling speed will produce a uniform temperature as it fills into the cavity. If the filling rate is slow, it may cause defects at certain thin part due to rapid cooling when the first material enters the cavity. Incomplete fill, lamination and warpage are the examples of defects due to slow speed of injection, especially for polypropylene and its copolymers because it has a relatively high crystalline melting temperature and solidify quickly in the cavity (Bauccio et al., 1994).

The thickness of a component usually affects the processing conditions in injection moulding. In this case, Table 2.4 proposed certain range of condition in the injection moulding process, specifically for polypropylene artefacts, based on various thicknesses.

Table 2.4: Certain ranges of condition for polypropylene artefacts (Bauccio et al., 1994).

<table>
<thead>
<tr>
<th>Moulding Conditions</th>
<th>Thickness of sections</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1.6 mm</td>
</tr>
<tr>
<td>Rear cylinder temperatures (°C)</td>
<td>193-216</td>
</tr>
<tr>
<td>Middle cylinder temperatures (°C)</td>
<td>204-232</td>
</tr>
<tr>
<td>Forward cylinder temperatures (°C)</td>
<td>216-249</td>
</tr>
<tr>
<td>Nozzle temperatures (°C)</td>
<td>193-216</td>
</tr>
<tr>
<td>Melt temperatures (°C)</td>
<td>204-249</td>
</tr>
<tr>
<td>Mould coolant temperatures (°C)</td>
<td>10-27</td>
</tr>
<tr>
<td>Hydraulic injection pressure (MPa)</td>
<td>4-10</td>
</tr>
<tr>
<td>Typical cycle time (s)</td>
<td>5-10</td>
</tr>
<tr>
<td>Plunger forward time (s)</td>
<td>15-25</td>
</tr>
<tr>
<td>Total cycle time (s)</td>
<td>15-25</td>
</tr>
<tr>
<td>Shrinkage (%)</td>
<td>1-2</td>
</tr>
</tbody>
</table>

The thickness varies from 1.6 mm to 6.4 mm. Based on the details in Table 2.4; it shows that the melt temperature was reduced by the additional of thickness. With regard of quality matters, variables such as injection speed, pressure, clamping
pressure, melt temperature, mould temperature and cycle time need to be monitored (Bauccio et al., 1994).

Several previous studies have been used as references for the effects of the injection moulding processing condition, as stated in Table 2.5. For example, to support the statement saying that temperature and pressure are the major parameter setting that influence the output of this process, previous researchers (Huang & Tai, 2001) have found that the order of the influence of process parameters on the warpage of injected parts are first, packing pressure, followed by mould temperature; melt temperature, packing time and cooling time. In addition, upon the findings, the effect of interaction between mould temperature and melt temperature could not be ignored (Huang & Tai, 2001)

Table 2.5: Previous study about the effects of processing condition

<table>
<thead>
<tr>
<th>Author (Year)</th>
<th>Response</th>
<th>Processing conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>(Huang &amp; Tai, 2001)</td>
<td>Warpage</td>
<td>Packing pressure</td>
</tr>
<tr>
<td>(Fung et al., 2003)</td>
<td>Yield stress and elongation</td>
<td>Melt temperature</td>
</tr>
<tr>
<td>(Nagaoka et al., 2005)</td>
<td>Core structure</td>
<td>Injection speed</td>
</tr>
<tr>
<td>(Song et al., 2007)</td>
<td>Part thickness</td>
<td>Metering size, injection rate.</td>
</tr>
</tbody>
</table>

Other researchers (Fung et al., 2003), have concluded that the most influential process factor and the most easily influenced mechanical properties can be rectified. It was found that the melt temperature was the most influential factor to mechanical properties of both yield stress and elongation. The yield stress was more influenced by melting temperature than the elongation. The sequence of controllable factors that affecting the yield stress was slightly different from the elongation, but the melt temperature was still the most important factor above all (Fung et al., 2003)

A research conducted by a group of Japanese scientist (Nagaoka et al., 2005) have studied about the effect of injection moulding parameters on the properties of polypropylene sandwich. This research considers the moulding conditions such as injection speed, cylinder temperature, and mould temperature were related to the mechanical properties of the sandwich moulding. In the case of single material sandwich moulding, injection speed seemed to play no significant role, even though it was clearly demonstrated that the core volume increases with injection speed. Thus,
the dormant or active role of injection speed depending on the material system has been highlighted (Nagaoka et al., 2005).

A study about thin wall injection moulding process can be a proper guideline for manufacturing polypropylene hinges artefacts. A research (Song et al., 2007) had examined the effects of injection process parameters on the moulding process for ultra-thin wall plastic parts. As for the solutions, by using the orthogonal of Taguchi method and numerical simulation, the influence of different process parameters such as injection rate, injection pressure, melt temperature, metering size and part thickness, in the moulding process for ultra-thin wall plastic parts were determined. Based on the results, the part thickness was the decisive parameter to the moulding. On the other hand, metering size and injection rate were the principal factors in the moulding process. The accelerating injection rate can increase the filling ratio. Melt temperature and injection pressure were the secondary factors, however, higher melt temperature and injection pressure were also essential in the moulding process (Song et al., 2007).

Therefore; in summary, the part thickness, injection rate, melt temperature, packing pressure, filling time and screw speed are some of the important factors that need to be considered when performing an injection moulding process especially for thin layer parts such as an integral hinge.

2.1.5 Optimisation method

The quality of the injection mouldings depends on the material characteristics, the mould design and the process conditions. Taguchi Optimisation Method is one of the popular methods used to determine the effect and the optimise process conditions in order to optimise part quality such as shrinkage, warpage and strength. This method is a statistical method, which developed by Dr. Genichi Taguchi to improve the quality of manufactured goods. This method also can be applied in engineering, biotechnology, marketing and advertising. Taguchi methods have been used widely in engineering analysis to optimise the performance characteristics through the setting of design parameters. Taguchi method is also a strong tool for the design of high quality systems. To optimise designs for quality, performance, and cost, Taguchi Optimisation Method presents a systematic approach that is simple and effective (Taguchi et al., 2005).
Part design, mould design, machine performance and processing conditions are some of the factors that will affect the quality of moulded parts. During production, quality characteristics may deviate due to drifting or shifting of processing conditions caused by machine wear, environmental change or operator fatigue. Determining optimal process parameter settings critically influences productivity, quality, and cost of production in the plastic injection moulding industry. Previously, production engineers used either trial-and-error method or Taguchi’s parameter design method to determine optimal process parameter settings for injection moulding. Yadav et al. (2012) have made a review regarding the recent research in designing and determining process parameters of injection moulding. A number of research works based on various approaches have been performed in the domain of the parameter setting for injection moulding. These approaches, including mathematical models, Taguchi method, artificial neural networks (ANN), fuzzy logic, case based reasoning (CBR), genetic algorithms (GA), finite element method (FEM), nonlinear modelling, response surface methodology, linear regression analysis, Grey rational analysis and principal component analysis (PCA) were described. The strength and the weakness of each individual approaches have been discussed with the conclusions of the potential research in determining process parameters for injection moulding.

Many published studies have emphasized the implementation of the Taguchi Optimisation Method in improving the warpage in the injection moulded part (Gao & Wang, 2008; Ozcelik & Sonat, 2009; Tang et al., 2007) as well as a sink index (Erzurumlu & Ozcelik, 2006) and weld lines (Othman et al., 2012; Ozcelik, 2011; Xie & Ziegmann, 2011).

To quote an example of Taguchi Optimisation Method, a research Oktem et al.,(2007) has been conducted regarding how to reduce warpage problem which was related to the shrinkage variation, depended on process parameters during production of thin-shell plastic components for an automobile part. The results of this research showed that warpage and shrinkage were improved by 2.17% and 0.7% respectively. It can be clearly inferred from this conclusion that Taguchi Optimisation Method is sufficient to solve the warpage problem with shrinkage for thin-shell plastic components of or that part (Oktem et al., 2007).

Another example is mentioned in the paper published by researchers (Erzurumlu & Ozcelik, 2006), consist of minimisation of the warpage and sink index in terms of the process parameters of the plastic parts have different rib cross-section
types, and rib layout angle, via Taguchi Optimisation Method. This method was used by exploiting mould analyses based on three level factorial designs. Orthogonal arrays of Taguchi, the signal to noise ratio, the analysis of variance were utilized to find optimal levels and the effect of process parameters on warpage and sink index (Erzurumlu & Ozcelik, 2006).

There was a research about the influence of injection parameters and mould materials on mechanical properties of acrylonitrile–butadiene–styrene (ABS) in plastic injection moulding (Ozcelik et al., 2010). The study was about optimising the effect of injection parameters such as melt temperature, packing pressure, cooling time and injection pressure towards the mechanical properties of mouldings. Signal to noise ratio for mechanical properties was calculated and the effect of the parameters on mechanical properties was determined using the analysis of variance (ANOVA). The regression analysis was also deployed to create linear mechanical models (Ozcelik et al., 2010).

Another study about the application of back propagation (BP) neural–network model to predict warpage and optimize the injected plastic parts has been developed based on key process variables, including mould temperature, melt temperature, packing pressure, packing time and cooling time during plastic injection moulding. The approach uses a BP neural network trained by the input and output data obtained from the finite element simulations which were performed on MoldFlow software platform. The product selected for this study is an automobile glove compartment cap. Trained by the results of finite element simulations conducted by orthogonal experimental design method, the prediction system applies a mathematical equation mapping the relationship between the process parameter values and the warpage value of the plastic. It has been proved that the prediction system has the ability to predict the warpage of the plastic within an error range of 2%. Process parameters have been optimised with the help of the prediction system. Meanwhile, energy consumption and production cycle were also taken into consideration. The optimised warpage value is 1.58 mm, which is shortened by 32.99% compared to the initial webpage result 2.358 mm. And the cooling time has been decreased from 20 s to 10 s, which will greatly shorten the production cycle. The final product can satisfy with the matching requirements and fit the automobile glove compartment well (Yin et al., 2010).

In order to obtain more accurate prediction of the service performance and service life of polymers and the optimisation of processing parameters, a novel on-
line testing equipment had been developed (Wang et al., 2009). The results of this project were compared with those obtained by the confining fluid technique. The Pressure-Volume-Temperature (P-V-T) curves were consistent, which proved that the new on-line measurement is feasible. The repeatability of the P-V-T measurement was within 0.3%. Consequently, a new P-V-T database of typical commercial materials could be established (Wang et al., 2009).

Some previous academics (Mehat & Kamaruddin, 2011) have studied the mechanical properties of products made from recycled plastic by utilizing the Taguchi Optimisation Method. By determining the optimal combination of factors and levels, the appropriate blending ratio of virgin and recycled plastic can be evaluated from the mechanical performance exhibited by the compound. The results reveal that the product made of 25% recycled polypropylene and 75% virgin polypropylene exhibits a better flexural modulus compared to the virgin form. The same product exhibits a 3.4% decrease in flexural strength.

Several scientists (Prashantha & Soulestin, 2009) have focused on the effect of multi-walled carbon nanotube (MWNT) addition on shrinkage and warpage properties of polypropylene injection mouldings before and after annealing. On the other hand, a research (Kramschuster et al., 2005) had presented the effects of processing conditions on the shrinkage and the warpage behaviour of a box-shaped, polypropylene part using conventional and microcellular injection moulding. In this research, after the injection moulding process reached steady state, the results show that the supercritical fluid content and the injection speed affect the shrinkage and warpage of microcellular injection moulded parts the most, whereas pack/hold pressure and pack/hold time have the most significant effect of these defects (Kramschuster et al., 2005).

A research (Rajesh & Soulestin, 2012) had successfully defined the effect of injection moulding parameters on nanofillers dispersion in masterbatch based polypropylene-nanoclay nanocomposites. The effect of injection moulding parameters (screw rotation speed, back pressure, injection flow rate and holding pressure) on the nano filler dispersion of melt mixed polypropylene-nanoclay nanocomposites was investigated. The nanocomposites containing 4 wt. % of clay were obtained by dilution of a polypropylene-nanoclay masterbatch into a polypropylene matrix. The evaluation of the dispersion degree was obtained from dynamic rheological measurements. The storage modulus and complex viscosity exhibit significant dependence on the injection moulding parameters. Polypropylene-nanoclay nanocomposite moulded using more
severe injection parameters (high shear and long residence time) displays the highest storage modulus and complex viscosity, which illustrates the improved dispersion of clay platelets. This better dispersion leads to better mechanical properties, particularly higher Young modulus, tensile strength and unmatched impact strength. The major individual parameter identified is the injection flow rate, whose increase improves nanoclay dispersion. The combination of high back pressure and high screw rotational speed is also necessary to optimize the dispersion of clay nano platelets (Rajesh & Soulestin, 2012).

Since polymer materials exhibit extremely convoluted properties, the complexity of the moulding process has become very tough to attain desired part properties (Mehat et al., 2014). However, most research related to optimisation only focus on a single response or quality characterstic. In reality, determining the superlative process parameters and focusing on multi-responses are intricate tasks, but are generally required (Khan et al., 2010). Therefore a constitutive approach to solve multi-response problems using the combination of single responses through the Taguchi Optimisation Method was needed to solve this problem.

Table 2.6 shows researchers related to multiple response optimisations. Previous researchers (Annicchiarico et al., 2013; Antony & Anand, 2006; Fung & Kang, 2005; Khan et al., 2010; Mehat & Kamaruddin, 2011; & Zhou & Turng, 2007) have published several articles related to multiple responses optimisation.

To quote an example, a research (Mehat & Kamaruddin, 2011); had used the accumulation of signal to noise ratio to define the optimum processing parameter in their research. By combining the responses to be studied concurrently, the approach seems to have improved the flexural modulus and strength of the produced part using recycled plastic compared with the single response approach.

Therefore, the literatures have clearly shown that Taguchi Optimisation Method has been used to determine optimum processing conditions for injection moulding analysis and it was successfully proven. The author will use this method in this research to achieve the objectives whereby optimise the processing condition and to generate regression models for producing integral hinges made from polypropylene nanoclay nanocomposites, via injection moulding process.
Table 2.6: Summary of multiple response optimisations.

<table>
<thead>
<tr>
<th>Author (Year)</th>
<th>Method of Optimisation</th>
<th>Findings</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fung &amp; Kang, 2005</td>
<td>Taguchi, Principal Component Analysis</td>
<td>Optimum combination of process factor and the most influential parameter for friction properties,</td>
</tr>
<tr>
<td>Antony &amp; Anand, 2006</td>
<td>Taguchi, Neuro Fuzzy Model</td>
<td>Optimised responses for a double sided surface mount technology of the electronic assembly process.</td>
</tr>
<tr>
<td>Zhou &amp; Turng, 2007</td>
<td>Gaussian process approach, multi objective genetic algorithm</td>
<td>Optimised system and process conditions for minimizing injection pressure, volumetric shrinkage/warpage and cycle time.</td>
</tr>
<tr>
<td>Khan et al., 2010</td>
<td>Grey Relational Analysis and Principle Component Analysis</td>
<td>Optimal combination of parameter and weighting value of tensile, compressive and flexural strength.</td>
</tr>
<tr>
<td>Mehat &amp; Kamaruddin, 2011</td>
<td>Taguchi</td>
<td>Optimum processing condition for flexural modulus and strength.</td>
</tr>
<tr>
<td>Annicchiarico et al., 2013</td>
<td>Design of experiment</td>
<td>Significant factors for shrinkage and part mass.</td>
</tr>
</tbody>
</table>

2.2 Polypropylene- nanoclay nanocomposites

Recently polypropylene nanocomposites have attracted much attention due to advances in the methods of their manufacture. Great expectation has been directed to this material and indeed it is not an exaggeration to suggest that an investigation into polypropylene nanocomposites have occupied the major proportion of those studies relating to polymer nanoclay nanocomposites (Mittal, 2009). Polymer nanocomposites consisting of a polymer and layered silicate, whether it was modified or not, have exhibited a remarkably improved mechanical and material properties when compared to those pristine polymers. These improvements include a higher modulus, increased in strength and better heat resistance. Recently polypropylene-nanoclay nanocomposites have increased great attention due to their improvement in properties such as high modulus, increased strength and heat resistance, decreased gas permeability and flammability. Efforts have been established nowadays, whereby many researchers have carried out more experiments concerning the combination of

By adding small amounts of nanoclay into polypropylene, both its melt characteristic (processabilities) and solid characteristics (product properties) are greatly altered. The main characteristic is that the non-Newtonian flow behaviour at low shear rates becomes more notable and shows a yield flow feature. A second plateau feature appears at low angular frequencies, which is more of a solid like characteristic. Taken together, these melt characteristic will affect the flow processabilities of the polymer (Mittal, 2009).

2.2.1 Micro mechanism, structure, morphology and properties.

In terms of the micro mechanism of plastic deformation during impact loading of polypropylene-nanoclay nanocomposites, researchers (Yuan & Misra, 2006) have examined and compared the condition with the unreinforced polypropylene under identical conditions of processing to underscore the determining role of clay. The addition of clay to polypropylene increases the impact strength in the temperature range of 0 to +70 °C. The enhancement of toughness on reinforcement of polypropylene-nanoclay is associated with change in the primary mechanism of plastic deformation from crazing and vein-type in neat polypropylene to micro void-coalescence-fibrillation process in the nanocomposite (Yuan & Misra, 2006).

As observed by Misra et al., (2010) the evolution of structure and phases during pressure-induced crystallization of polymers had been summarised. The polymer containing a dispersion of nanoparticles, in the pressure range of 0.1–200 MPa. The model material for nanoparticles is nanoclay and the model polymer is polypropylene, which can potentially form several crystalline phases. While the phase selection in polypropylene is dictated by pressure and temperature, however, the introduction of nanoparticles alters the nucleation and growth of phases via nanoparticles interface driven evolution. To delineate and separate the effects of applied crystallization pressure from nanoparticles effects, a relative comparison is made between neat polypropylene and polypropylene containing a dispersion of nanoclay under similar experimental conditions. The significant finding of the research was that nanoclay interacts with the host polypropylene in a manner such that it alters the structural
morphology of [alpha]- and [gamma]-crystals of polypropylene. Furthermore, nanoclay promotes the formation of [gamma]-phase at ambient pressure, suggesting its role as structure and morphology director in the stabilization of the less accessible [gamma]-phase, and with the possibility of epitaxial growth that enhances toughness. The equilibrium melting point measurements point to thermodynamic interaction between polypropylene-nanoclay, which is supported by the change in glass transition temperature. Thus, the two components, nanoclay and pressure, together provide a unique opportunity to tune hierarchical structures and phase evolution, which has significant implication on physico-chemical and mechanical properties (Misra et al. 2010).

Significant differences in the microstructure occur when the polypropylene matrix dispersed with 4wt. % and 8wt. % nanoclay. In neat polypropylene, crystals nucleate in low to moderate crystallization pressure range of (~0.1–60 MPa), while -phase nucleates at a crystallization pressure of 60–200 MPa. In contrast to neat polypropylene, the presence of nanoclay in the polymer matrix, promotes the formation of phase at an ambient crystallization pressure of 0.1MPa. Nanoclay and pressure-induced crystallization governs phase evolution and microstructure of Polypropylene and is associated with change in the thermodynamic and physical properties such as equilibrium melting point and glass transition temperature. This change in equilibrium melting point and glass transition temperature has pointed the thermodynamic interaction between polymer nanoparticles and nanoparticles interface driven microstructural evolution. However, the effect of nanoparticles is less dramatic when nanoclay content is increased from 4wt. % to 8wt. %, implying that the effect maximizes at 4 wt. % nanoclay (Yuan et al. 2010).

A paper presents the results of a current study concerning the mechanical properties of a polypropylene binder resin, enhanced by nanoclay filler. The study was centred on the potential benefits obtained by the addition of especially surface treated nanoclay on the stiffness, toughness and also on static and fatigue strength. Specimens were produced by an injection moulding process with 3% by weight of nanoclay. Surface treatment of the nanoclay promotes a tendency to increase stiffness and tensile strength in comparison with composites filled by both commercial nanoclay and unfilled material. Nanoclay filling significantly improves absorbed impact energy in comparison with unfilled materials. Fatigue analysis shows that the materials tested in
the present work exhibit high strain accumulation and stress release. Filled material show lower fatigue performance than unfilled polymer (Ferreira et al. 2011).

Observations are reported on isotactic polypropylene/organically modified nanoclay hybrids in tensile cyclic tests at room temperature. A pronounced effect of nanofillers is demonstrated: reinforcement of polypropylene with 1 wt. % of clay results in reduction of maximum and minimum strains per cycle by several times and growth of number of cycles to failure by an order of magnitude. To rationalize these findings, a constitutive model is developed in cyclic viscoelastoplasticity of polymer nanocomposites. Adjustable parameters in the stress–strain relations are found by fitting experimental data in relaxation tests and cyclic tests (16 cycles of loading–unloading). It is demonstrated that the model correctly predicts growth of maximum and minimum strains per cycle with the number of cycles and can be applied to predict fatigue failure of nanocomposites. The highlights of this research are the reinforcement strongly reduces ratcheting strain in polypropylene-nanoclay hybrids, a constitutive model is derived for lifetime evaluation under fatigue conditions and only 1 wt. % of nanoclay are sufficient to increase the number of cycles to failure by an order of magnitude (Drozdov, 2012).

A study about the morphology, orientation, as well as the mechanical and barrier properties of monolayer and multilayer nanoclay filled polypropylene obtained from the film blowing process were investigated. A significant alignment of nanoclay along the flow direction was observed from scanning electron microscopy images of the cross-section of the etched monolayer and trilayer films. The orientations of polypropylene crystallites and clay platelets as well as the extent of intercalation and exfoliation were analysed using a wide angle X-ray diffraction. The crystalline structure formed in polypropylene alone was such that the b-axis oriented in the normal and transverse directions (ND and TD, respectively) and the c-axis aligned in the machine direction (MD). The addition of clay changed the orientation of the b-axis to ND, enhanced the a-axis orientation in MD, and also improved the c-axis alignment along MD. Moreover, the 001 plane (normal to platelets plane) of the nanoclay lay into ND drastically. With the incorporation of the clay tactoid, the Young’s modulus was enhanced by 25—40%, the tensile strength remained almost unchanged, and the elongation at break along TD decreased by more than 70% of all the films. The permeability to oxygen and tear resistance along MD and TD were slightly reduced by the presence of nanoparticles and the percentage of nanofillers studied. The haze of
the nanocomposite films increased upon the presence of clay particles, except in the case of low clay contents of 1.5 and 2.5 wt.% (Tabatabaei & Ajji, 2011).

### 2.2.2 Preparation methods

The preparations of polypropylene-nanoclay nanocomposites are divided into three main methods according to the starting materials and processing techniques. The methods are the intercalation of polymer or pre-polymer from solution, in situ intercalative polymerization method and melt intercalation method (Pavlidou & Papaspyrides, 2008).

The intercalation of polymer or pre-polymer from solution is the first method found in preparing the polypropylene-nanoclay. This method is based on a solvent system in which the polymer or pre-polymer is soluble and the silicate layers are swellable. The layered silicate is first swollen in a solvent, such as water, chloroform, or toluene. When the polypropylene-nanoclay solutions are mixed, the polymer chains intercalate and displace the solvent within the interlayer of the silicate. Upon solvent removal, the intercalated structure remains, resulting in polypropylene-nanoclay nanocomposite (Pavlidou & Papaspyrides, 2008).

The in situ intercalative polymerization method is the second method of preparing the polypropylene-nanoclay. This method was successfully applied by manipulating the ability of soluble metallocene catalysts to intercalate inside silicate layers, and to promote the coordination polymerization of propylene. A synthetic hectorite was first treated with methylaluminoxane in order to remove the acidic protons, and to prepare the interlayer spacing for receiving the transition metal catalyst. Another method was also discovered by making the layered silicate swollen within the liquid monomer so the polymer formation can occur between the intercalated sheets (Pavlidou & Papaspyrides, 2008).

The third method of polypropylene-nanoclay preparation is through melt intercalation. Recently, this technique has become the standard for the preparation of polypropylene-nanoclay nanocomposites (Sinha Ray & Okamoto, 2003). During polymer intercalation from solution, a relatively large number of solvent molecules have to be desorbed from the host to accommodate the incoming polymer chains. The desorbed solvent molecules gain one translational degree of freedom, and the resulting
entropic gain compensates for the decrease in conformational entropy of the confined polymer chains. This method involves annealing, statically or under shear, and a mixture of the polypropylene-nanoclay above the softening point of the polymer. While annealing, the polymer chains diffuse from the bulk polymer melt into the galleries between the silicate layers. A range of nanocomposites with structures from intercalated to exfoliate can be obtained, depending on the degree of penetration of the polymer chains into the silicate galleries. So far, experimental results indicate that the outcome of polymer intercalation depends critically on silicate function and constituent interactions. This method has great advantages over either in situ intercalative polymerization or polymer solution intercalation such as this method is environmentally nonthreatening due to the nonexistence of organic solvents. It is also compatible with current industrial process, such as extrusion and injection moulding. The direct melt intercalation was highly specific for this polymer, leading to new hybrids that were previously inaccessible. Last but not least, the advantage of this method is the absence of a solvent, which made direct melt intercalation as an economic process for industries from a waste perspective (Sinha Ray & Okamoto, 2003).

A study (Albdiry & Yousif, 2013) had been conducted to determine the difference of polymer-nanoclay processing techniques. The researchers have performed the review in order to study the relationship between the techniques and the final characteristics and properties of polymer-nanoclay nanocomposites, in terms of the final structure formation, rheological perfection, thermo-mechanical and thermal properties. Thermodynamic and physical properties such as glass transition temperature, equilibrium melting point and crystallization temperature were also discussed in this review. The review concluded that the altering process technique (type and/or parameters) highly influences the final nanostructure morphology as well as the thermodynamic and mechanical properties of nanoclay/polymer composites (Albdiry & Yousif, 2013).

Therefore, based on these findings, melt intercalation via twin screw compounder was chosen as the preparation method for polypropylene-nanoclay in this research.
REFERENCES


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